

# BULLETIN

## OF THE INSTITUTE OF METALS

VOLUME 2

MAY 1954

PART 9

### INSTITUTE NEWS

#### Autumn Meeting in Switzerland

By invitation of the Société Suisse des Constructeurs de Machines and the Association Suisse pour l'Essai des Matériaux, the 1954 Autumn Meeting of the Institute will be held in Switzerland from Monday, 6 to Tuesday, 14 September, inclusive. From Monday, 6 to Saturday, 11 September the meeting will be in Zürich; on the Saturday members and their ladies will go by special train to Montreux, where the meeting will conclude.

Full particulars will be published at an early date. Members hoping to be present are requested to be prepared to reserve hotel accommodation not later than June. Travel and hotel arrangements will be in the hands of Thos. Cook and Son, Ltd.

#### Honorary Treasurership

Because of the grave illness (and subsequent death) of Colonel Sir Paul Gueterbock, Managing Director of Capper Pass and Son, Ltd., the Honorary Treasurer, Mr. E. H. JONES, who is a Director of the Company, has had to undertake heavy additional business responsibilities. In consequence, he has felt bound to tender his resignation as Honorary Treasurer. The Council has accepted Mr. Jones's resignation with much regret, and has expressed its warm thanks to him for the services he has rendered to the Institute during his period of office.

Under its powers in Article 28, the Council has elected Mr. J. C. COLQUHOUN, M.B.E., Chairman and Managing Director, The Manganese Bronze and Brass Co., Ltd., as Honorary Treasurer. He takes office on 1 May 1954 and will present himself for re-election at the next election of Council.

#### Chairmen of Committees

The Council has appointed the following Chairmen of Committees for 1954-55:

*Finance and General Purposes Committee.*—Mr. E. H. JONES.

*Local Sections Committee.*—Dr. L. B. PFEIL.

*Medal Committee.*—The PRESIDENT.

*Metal Physics Committee.*—Professor G. V. RAYNOR.

*Metallurgical Engineering Committee.*—Mr. W. J. THOMAS.

*Nominations Committee.*—The PRESIDENT.

*Publication Committee.*—Mr. CHRISTOPHER SMITH.

#### W. H. A. Robertson Medal and Premium for 1953

No award is being made of the W. H. A. Robertson Medal and Premium for 1953.

#### Students' Essay Prize Competition, 1953

The Students' Essay Prize Competition for 1953 has been won by Mr. DEREK HULL (Student Member), of the Department of Metallurgy, University College, Cardiff, for an essay on "The Characteristics of the Martensite Transformation". The essay has been recommended for publication in the *Bulletin*.

#### Revised Edition of "The Structure of Metals and Alloys"

(Monograph No. 1)

This well-known book by Dr. W. Hume-Rothery was first published in 1936, when it achieved an immediate success, and has since been reprinted seven times. When first written, it contained a considerable amount of original matter and was intended for both research workers and University students. In the intervening years the subject matter has been incorporated into University degree courses, for which the book has become a standard text. Most of the principles outlined in 1936 have stood the test of time, but the tremendous output of research on the structure of metals and alloys has led to a demand for a thorough revision, and a new edition has now been written in collaboration with Professor G. V. Raynor.

The whole text has been revised and brought up to date, with considerable expansion of the parts dealing with solid solutions and intermediate phases in alloys. Completely new sections have been written on ternary alloys and on alloys of iron, whilst the part dealing with imperfections in crystals has been greatly extended and now includes a comprehensive account of the ideas of dislocation theory.

The description throughout has been kept at an elementary level, and can be understood without a knowledge of higher mathematics, or of crystallographic notations. In this form the book should be invaluable to all those who, in the widest sense of the term, call themselves students of metallurgy, and it should also be of great value to physicists, chemists, and engineers.

The new edition, containing 370 pages and 8 plates, is being published this month. It is available from the Institute or through booksellers, price 35s. (or \$5.50), post free. Members may obtain one copy at the special reduced price of 17s. 6d. (\$3.00), post free.

## PERSONALITIES

### Election of Members

The following 14 Ordinary Members, 3 Junior Members, and 21 Student Members were elected on 30 March 1954:

#### *As Ordinary Members*

- VAN DEN BERGH, Antonius Wilhelmus Josephus, Ing., Chief Metallurgist and Chief of the Laboratory for Testing and Research of Materials, N. V. Kromhout Motoren-fabriek v.h.D. Goedkoop Jr., Ketelstraat, Amsterdam (N), Netherlands.
- BOTHMANN, Hans, Dipl.-Ing., Geschäftsführer, Westfälische Leichtmetallwerke G.m.b.H., Nachrodt, Kreis Altena (Westf.), Germany.
- CAPE, A. Tregoning, A.R.S.M., Vice-President in Charge of Research and Development, Superweld Corporation, 6840 Vineland Avenue, North Hollywood, Calif., U.S.A.
- CASTLEDINE, Walter, Production Engineer, Salopian Engineers, Ltd., Prees, Whitchurch, Shropshire.
- DAS GUPTA, Annadananda, M.Sc., Works Manager, Rolling Mills, Indian Aluminium Company, Ltd., 375A Grand Trunk Road, Belurmath P.O., Howrah, West Bengal, India.
- DEYSINE, Jean, Ing. Civil des Mines, Directeur Technique, Société Française des Métaux et Alliages Blancs, 76 Boulevard Anatole France, Saint Denis (Seine), France.
- ERBSLÖH, Paul Günther, Geschäftsführer und Teilhaber, Julius und August Erbslöh, Metallwalz- und Presswerke, Berlinerstrasse 29-35, Wuppertal-Barmen, Germany.
- EVANS, Cyril James, Heat-Treatment Engineer, The General Electric Co., Ltd., Magnet House, Kingsway, London, W.C.2.
- FIELDING, John, A.M.C.T., A.M.I.Mech.E., Chief Metallurgist, Research Department, A. V. Roe and Co., Ltd., Greengate, Middleton, Manchester.
- GÜRTLER, Gustav, Dr.-Ing., Technical Managing Director, Aluminium-Zentrale E.V., Jägerhofstrasse 26/29, Düsseldorf, Germany.
- HORGAN, Michael O'Connor, T.D., M.Sc., M.I.E.E., Sales Engineer, Process Heating Department, The General Electric Company, Ltd., Magnet House, Kingsway, London, W.C.2.
- KOOPMANN, Wilhelm Eduard Karl Robert, Ministerialrat a.D., Dipl.-Ing., Technischer Direktor, Vereinigte Deutsche Metallwerke A.G., Zweigniederlassung Basse und Selve, Werdohlerstrasse 62, Altena (Westf.), Germany.
- REYNOLDS, James E., D.Sc., Battelle Memorial Institute, 505 King Avenue, Columbus 1, Ohio, U.S.A.
- SCOBLE, Gerald Raymond, Branch Manager, Industrial Metals, Ltd., 15 Courtenay Place, Wellington, New Zealand.

#### *As Junior Members*

- ANASTASSIOU, Basil D., Graduate of National Technical University of Athens. (mail): Ierosolymon St. 18, Athens, Greece.
- CROSS, John Philip, Assistant Metallurgist, Johnson, Matthey and Co., Ltd., Exhibition Grounds, Wembley, Middx.
- THOMAS, John Stuart, B.Sc., Research Metallurgist, Engine Division, The Bristol Aeroplane Company, Filton House, Bristol.

#### *As Student Members*

- BONE, David Charles, Student of Metallurgy, Royal School of Mines, London, S.W.7.
- CHASTON, Ian Robert Malcolm, Student of Metallurgy, Royal School of Mines, London, S.W.7.

- COX, David Smedley, Student of Metallurgy, Royal School of Mines, London, S.W.7.
- DAY, Joseph George, Metallurgist, Research Laboratories, Johnson, Matthey and Co., Ltd., Wembley, Middx.
- DAY, Ralph Victor, Student of Metallurgy, Royal School of Mines, London, S.W.7.
- DEIGHTON, Malcolm, Undergraduate, Department of Metallurgy, King's College, University of Durham, Newcastle-on-Tyne.
- EVANS, Peter Rees Vaughan, B.Sc., Research Student, Department of Metallurgy, University College of Swansea.
- FORSCHER, Richard, Student of Metallurgy, Royal School of Mines, London, S.W.7.
- HALLETT, (Miss) A. R., Student of Metallurgy, University of Manchester.
- HOLLAND, James Read, B.S., Postgraduate Student in Physical Metallurgy, University of Sheffield.
- NICHOLAS, Michael George, Undergraduate, Department of Industrial Metallurgy, University of Birmingham.
- NORREYS, John Jephson, Student of Metallurgy, Royal School of Mines, London, S.W.7.
- SMITH, Antony Burwood, Student of Metallurgy, Royal School of Mines, London, S.W.7.
- SMITH, Donald Peter, Student of Metallurgy, Royal School of Mines, London, S.W.7.
- STUART, George R., B.Sc., A.R.T.C., Assistant Metallurgist, Henry Wiggin and Company, Ltd., Thornliebank, Glasgow.
- TAYLOR, Roy, Undergraduate, Department of Metallurgy, University of Manchester.
- TREE, Frank, Student of Metallurgy, Royal School of Mines, London, S.W.7.
- WATKINS, Alan John, Undergraduate, Selwyn College, Cambridge.
- WEBB, Watt Wetmore, B.S., Graduate Fellow, Room 8-101, Massachusetts Institute of Technology, Cambridge 39, Mass., U.S.A.
- WILKINSON, Donald Hugh, Student of Metallurgy, Royal School of Mines, London, S.W.7.
- WILLIAMS, Bruce Oswald Bernard, Undergraduate, Pembroke College, Cambridge.

## PERSONALITIES

### Mr. J. C. Colquhoun (Honorary Treasurer)

James Clifton Colquhoun was born in 1893 in Clifton, Arizona, U.S.A., where his father, James Colquhoun, at that time was President of the Scottish-owned Arizona Copper Company, later acquired by the Phelps Dodge Corporation. His education at Trinity College, Glendale, and at Caius College, Cambridge, where he was reading for a Natural Science Tripos was interrupted by World War I.

After that war, in which he was mentioned in despatches and awarded the M.B.E., he returned to the copper smelters of Arizona for a brief period and later studied at the Royal School of Mines, London, until offered a position by Sir Cecil L. Budd, K.B.E., then Joint Managing Director of The British Metal Corporation. His business appointments since have been entirely with companies in which The British Metal Corporation held an interest, namely: Messrs. Vivian, Younger and Bond, Ltd., as Director; The Cornish Tin

## PERSONAL NOTES

Smelting Co., Ltd., as Director and Local Manager; The Manganese Bronze and Brass Co., Ltd., of which he is the present Chairman and Managing Director; The Greenside



Lead Mine on Lake Ullswater, as Director and Chairman for a time during the last war; and Lightalloys, Ltd., of which he is Chairman.

Mr. Colquhoun joined the Institute in 1924.

## PERSONAL NOTES

MR. A. C. DRUMMOND has been appointed General Manager and Secretary to the Lead Development Association. For the past seven years, Mr. Drummond has been Secretary of the Lead Industries Development Council, which has become the Lead Sheet and Pipe Council, an associate member of the Lead Development Association.

DR. A. R. ENTWISTLE has been appointed the first United Steel Companies' Research Fellow in Metallurgy at Sheffield University.

MR. S. FOLDES has left the Institute for the Study of Metals, Chicago, to take up a post as metallurgist with the Beryllium Corporation, Reading, Pa., U.S.A.

MR. O. P. HARTREE has left the Bristol Aeroplane Co., Ltd., Engine Division Laboratories, to become a Research Metallurgist in the Physical Metallurgy Section of the A.E.I. Research Laboratories, Aldermaston, Berks.

MR. O. J. HINETT is now Metallographer with Joseph Sankey and Sons, Ltd., Wellington, Salop.

MR. J. C. HOWARD has been made a Director of EFCO Engineering Co., Ltd.

DR. J. P. HUGO has taken a post in the Metallurgy Section of the Council for Scientific and Industrial Research, Pretoria, South Africa.

MR. A. E. JENKINS has been awarded the Ph.D. degree of Melbourne University.

MR. E. H. JONES has been appointed Joint Managing Director of Capper Pass and Son, Ltd., Bristol.

MR. H. D. JONES has left Dorman, Long and Co., Ltd., to take up an appointment with the Round Oak Steel Works, Ltd., Brierley Hill, Staffs.

LIEUT.-COLONEL G. MACWATERS has been appointed to the board of Capper Pass and Son, Ltd., Bristol.

MR. JOHN H. PARRY is now Lecturer in Metallurgy at the Ipswich School of Technology.

THE HON. J. F. A. ROCHE has been appointed Joint Managing Director of Capper Pass and Son, Ltd., Bristol.

MR. N. B. RUTHERFORD has left the British Non-Ferrous Metals Research Association and is now Metallurgist to Cam Gears, Ltd., Luton, Beds.

MR. D. B. SCOTT has left the American Can Co. to take a post as Metallurgist with Revere Copper and Brass, Inc.

MR. J. H. O. VARLEY has left Birmingham University to take up an appointment in the Metallurgy Division of the Atomic Energy Research Establishment, Harwell.

## Death

The Editor regrets to announce the death of:

COLONEL SIR PAUL GUETERBOCK, K.C.B., D.S.O., M.C., T.D., J.P., A.D.C., Past-President and a Fellow of the Institute, at his home George's Plot, Abbots Leigh, near Bristol, on 8 March after a prolonged illness. Sir Paul was Managing Director of Capper Pass and Son, Ltd.

Among those present at a Memorial Service in Bristol Cathedral on 12 March were The Duke of Beaufort (Lord Lieutenant of Gloucestershire), the Lord Mayor and the Sheriff of Bristol, the Vice-Chancellor of the University of Bristol, and representatives of the Institute of Metals, the Institution of Mining and Metallurgy, and numerous military and public organizations with which Sir Paul had been connected.

## OBITUARY

### Mr. H. B. Weeks

With the death of Mr. H. B. Weeks, which occurred on 20 February 1954, at St. Albans, at the age of 90, the Institute lost one of its oldest members.

Henry Bridges Weeks was born at Plumstead, London, and trained at the Royal Arsenal Science School. He was for a time Chief Chemist and Metallurgist in the Ordnance Department of India, before joining Vickers, Sons and Maxim, Ltd. (later Vickers, Ltd., and now Vickers-Armstrongs, Ltd.) at Barrow-in-Furness in 1898 as Chief Chemist and Metallurgist in a small laboratory. As the early work expanded under his guidance, a larger and well-equipped laboratory was built in 1914.

Mr. Weeks was perhaps best known in connection with the development of Duralumin. This alloy was, of course, originally made in Germany and, some time during the period 1908-10, Mr. Weeks was delegated on behalf of Vickers, Ltd., to go to Germany and investigate its potentialities. It is to his credit that he persuaded the directors of his firm to take up the world rights of Duralumin outside Germany, the

American rights being, at a later date, leased to an American firm. The initial development work was in the hands of Mr. Horace Clarke, then at Woolwich. Mr. Weeks was associated with Mr. Clarke in the large-scale production of Duralumin by James Booth and Co. (1915), Ltd., a firm set up for its manufacture under the control of Vickers, Ltd. As is well known, Duralumin soon assumed a very important place when its potentialities as a high-strength light alloy were realized, and Mr. Weeks acted as Consultant to James Booth and Co. for many years.

In 1923 he was transferred to London and, until his retirement about ten years later, he acted in a consultative capacity on chemical and metallurgical matters to the Vickers Group.

Mr. Weeks naturally had other activities of a chemical and metallurgical character, being a Fellow of the Royal Institute of Chemistry, a Member of the Iron and Steel Institute, and an Original Member of the Institute of Metals, on whose Council he served continuously from 1920 to 1938. He was Chairman of the Council of the British Cast Iron Research Association for 13 years from its inception, and he also served on several committees of the British Standards Institution.

Mr. Weeks's staff, in whom he always showed a kindly interest, had a high respect for his abilities, his clear mind and precise manner of expression being particularly admired. Among his main private pursuits was tennis, which he continued to play until he was about 80 years of age, and he was a keen supporter of the Furness Rugby Union Football Club.

His wife died in 1948, and he leaves a son and daughter and three grandchildren.

M. C. OLDHAM

### Mr. J. H. Wilson

It is with deep regret that we record the death of Mr. J. H. Wilson, an Original Member of the Institute, at his home at Church Stretton, on 9 February, aged 77.

James Howard Wilson was born at Golds Hill, West Bromwich, in 1876 and began his business career in the iron trade in 1895. In 1903 he took control of James Clews and Sons and in 1919 merged this company with Evered and Co., Ltd., Smethwick, of which he was Managing Director until 1942, when he relinquished this position to become Chairman.

A man of wide activities, Mr. Wilson was a J.P. for the County Borough of Smethwick and Chairman for several years of the Smethwick Conservative Association. He was a member of many trade associations, including the Brass and Copper Tube Association, the Cold-Rolled Brass and Copper Association, and the Non-Ferrous Metals Association, and he was instrumental in forming the Metallic Bedsteads Association. A wide circle of associates in the metal industry will grieve the loss of a respected and well-loved friend.

Mr. Wilson leaves a widow and seven children, of whom the second son, Mr. A. H. Wilson, is now Managing Director of Evered and Co., Ltd.

## JOINT ACTIVITIES

### Sir George Beilby Memorial Awards

At a meeting on 9 March 1954 the Administrators of the Sir George Beilby Memorial Fund considered the claims for an award from the Fund of candidates whose merits had been brought to their attention. The Committee was impressed

by the promise shown by several of these candidates, but concluded that none of them adequately fulfilled the conditions of the award, which is made "in recognition of continuous work of exceptional merit, bearing evidence of distinct advancement in science and practice".

It was decided, therefore, to make no award for 1953, but to let it be known that this decision should not discourage future applications by, or on behalf of, the present candidates or other investigators working on subjects relating to the special interests of Sir George Beilby, including problems connected with fuel economy, chemical engineering, and metallurgy.

## NEWS OF LOCAL SECTIONS AND ASSOCIATED SOCIETIES

### London Local Section

At a meeting of the Section held at 4 Grosvenor Gardens, London, S.W.1, on 4 March, Dr. J. P. DENNISON (Lecturer in Metallurgy at University College, Swansea) spoke on:

### High-Temperature Corrosion

In view of the wide field of the subject, the lecture was devoted to a survey of the behaviour of alloys likely to be used in the gas-turbine, with particular reference to the effect of sulphur in the atmosphere and to the heat-interchanger problem.

The lecturer said that economic considerations would probably require the larger land or marine gas-turbines to use relatively low-grade fuels, and that in certain residual oils the presence of vanadium or sodium in the ash had caused severe attack at temperatures as low as 600°C., owing to the formation of low-melting-point vanadate complexes. The solution of this problem appeared to lie outside the scope of the metallurgist, but the effect of sulphur, which had more frequently been encountered, had received much attention.

The presence of hydrogen sulphide in atmospheres where there was no free oxygen could cause severe attack, leading to the failure of almost all steels and nickel-base alloys which relied for their scaling resistance on the formation of protective oxides. Little trouble was normally encountered in the usual gas-turbine atmosphere, provided that complete combustion was achieved which resulted in the presence of 17-18% free oxygen in the atmosphere.

A study of the copper-base alloys available for use as heat-interchanger tubes had shown the copper-aluminium series to offer most promise, but industrial use of these alloys had failed to uphold the satisfactory results obtained in laboratory tests. This failure might be due in part to different conditions of exposure, particularly the possibility of exposure to atmospheres containing sulphurous gases at or near the condensation point, and in part to the initial surface condition of the material under test.

The main requirement for such an alloy appeared to be the formation and retention of a thin protective film during exposure; in this respect the addition of small amounts of titanium or zirconium to a single-phase copper-aluminium alloy had been shown to have considerable effect on the type of scale formed and also in improving the adherence of the latter under severe conditions of test.

# PRODUCTION AND PROPERTIES OF HIGH-PURITY NICKEL B7 POWDER \*

By PROFESSOR F. A. FORWARD,† B.A.Sc.

**SYNOPSIS:** *A description is given of a new process that has been developed to produce high-purity nickel powder from a Canadian ore. The process consists of a combined ammonia-leaching and hydrogen-reduction operation, which is outlined. By this means nickel powders can be produced to meet a wide variety of particle-size specifications. Possible uses are indicated.*

## Introduction

THE pure nickel used for preparing high-grade nickel alloys, sheet, tube, and other products is commonly supplied in the form of cut electrolytic cathodes, shot, or pellets. Electrolytic nickel usually contains 99.95+ % nickel + cobalt (of which 0.1–0.8 % is cobalt), less than 0.01 % sulphur, 0.01–0.03 % each of iron and copper, and traces of other impurities such as lead, arsenic, and antimony. By using special techniques, electrolytic nickel containing 99.98 % nickel + cobalt, of which 0.1–0.5 % is cobalt, can be produced. Nickel shot obtained by the Mond process is free from cobalt, copper, and sulphur, but may contain up to 0.1 % carbon, as well as a minor amount of iron.

It is the purpose of this paper to describe in general terms the techniques which have been used to produce nickel in powder form containing 0.10 % cobalt, about 0.01 % each of iron and sulphur, less than 0.005 % copper, spectrographic traces of lead, and no detectable amount of carbon, zinc, arsenic, antimony, or other impurity. The process referred to will be employed on a commercial scale in the Sherritt Gordon nickel refinery at Fort Saskatchewan, Alberta, which is starting operations early in 1954 and which is scheduled to produce nickel at the rate of 8500 tons/year.

The Sherritt Gordon mine at Lynn Lake, Manitoba, has a reserve of 14,000,000 tons of nickel ore, containing 1.225 % nickel and 0.618 % copper, with additional cobalt values. During the past five years, the mine has been opened up and equipped to produce 2000 tons of ore/day; this will be concentrated in a mill to produce a copper concentrate and about 235 tons/day of nickel-copper-cobalt sulphide concentrate containing: nickel 10–16, copper 1–2, cobalt 0.2–0.4, iron 30–40, sulphur 28–34, SiO<sub>2</sub>, &c. 8–14 %, and precious metals < 0.02 oz./ton.

The nickel concentrate is to be dried and shipped about 800 miles by rail to Fort Saskatchewan, where it will be treated by an ammonia-leaching process which has been developed in laboratory and pilot-plant operations carried out by the Sherritt Gordon Metallurgical Research Division in its Ottawa pilot plant during the past five years, at a cost of \$2,500,000. The Fort Saskatchewan refinery will include, in addition to the nickel section, a synthetic-ammonia plant and a crystallizing unit to produce ammonium sulphate fertilizer which will be recovered as a by-product.

## Ammonia-Leaching Process

The ammonia-leaching process, as carried out in the pilot plants, is relatively simple. In a small operation (3000 lb. concentrate/day), the concentrate is mixed with ammonia, air, and water and is agitated mechanically in horizontal autoclaves under a pressure of approximately 100 lb./in.<sup>2</sup>. The autoclaves are fed and discharged continuously and are so arranged, with filters and thickeners, that a two-stage continuous counter-current leach is effected.

The nickel, copper, and cobalt are dissolved and recovered in the pregnant liquor as amines, and, as the leach is basic, most of the sulphur is oxidized and appears in the pregnant liquor as sulphate, thiosulphate, thionate, and sulphamate ions. The iron is converted to insoluble hydrated ferric oxide, which, together with silicates and other insoluble materials, is separated from the leach liquors by filtration, washed, and discarded.

The pregnant liquor is boiled in continuous distillation units to remove and recover part of the ammonia and to decompose some of the thionates present. The decomposition of thionates produces sulphide ions that convert the copper present to copper sulphide, which is separated from the leach solution by filtration, leaving the nickel and cobalt amines, some free ammonia, ammonium sulphate, thiosulphate, thionate, and sulphamate in a copper-free solution. By suitable control of the copper-precipitation step, it is possible to reduce the copper from 8 g./l. in the pregnant liquor to less than 0.005 g./l. (usually 0.001 g./l.) in the copper-free solution, which contains at the same time about 45 g./l. nickel and 0.7 g./l. cobalt.

## Hydrogen-Reduction Process

In presenting the following description of the next stage of the process, i.e. the hydrogen-precipitation, the author lays no claim to its origination or development. The procedure for precipitating nickel from the leach solutions, using hydrogen under pressure, was suggested in the first place by the research workers and engineers of the Chemical Construction Corporation, who also carried out the early experimental work in the laboratory and in small pilot-plant autoclaves. During the past four years, the research and operating groups of the Sherritt Gordon Metallurgical Research Division have been responsible for solving the chemical and mechanical problems involved in developing the process to the point where it is

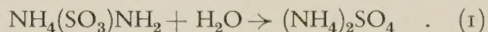
\* Lecture delivered before the London Local Section at the Royal School of Mines on 5 November 1953.

† Head of the Department of Mining and Metallurgy, University of British Columbia, Vancouver, Canada.

capable of maintaining uniform production of high-purity nickel metal from pilot-plant leach solutions with regularity and within easily controlled limits. The process as now operated in the pilot plant is the product of the ingenuity, systematic study, and careful prolonged work by scientists and engineers in both the groups mentioned, and it is to be hoped that, as soon as time is available, the men who are responsible for the development of the various phases of the project will present the details of their work much more fully than is possible in the present paper.

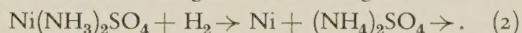
The primary purpose of the reduction process is to produce pure nickel and cobalt metal from the copper-free solution by using hydrogen at elevated temperature and pressure. If, however, the nickel metal is to be pure, some provision must be made to supply a highly purified solution to the reduction unit, and as the "end solutions" are evaporated to produce ammonium sulphate, means must also be provided to remove (and recover) from them the last traces of nickel and cobalt.

The purification of the copper-free solution has two principal objectives: (i) to oxidize unsaturated sulphur compounds such as thionate and thiosulphate to sulphate and (ii) to hydrolyse sulphamate to sulphate, with the purpose of preventing precipitation of sulphides on the surfaces of heat exchangers and pressure vessels or with the nickel powder during reduction. The initial oxidation is accomplished by heating the solution in a continuous, mechanically stirred autoclave at about 350° F. (175° C.) in the presence of compressed air at approximately 200 lb./in.<sup>2</sup> overpressure. The oxidized solution, maintained under pressure, is passed directly and continuously to a second autoclave, which is held at about 450° F. (230° C.), at which temperature the ammonium sulphamate hydrolyses to form ammonium sulphate according to the following reaction:



Solid impurities precipitated from the solution during the oxidation and hydrolysis stage are removed, by filtration, and the clarified solution is passed to a high-pressure holding tank.

The purified solution is then treated with hydrogen under pressure in a horizontal, mechanically stirred autoclave to precipitate nickel according to the following reaction:



Cobalt reacts in exactly the same way, but the operation can be carried out in such a manner that the nickel is precipitated preferentially, leaving the major portion of the cobalt in solution. The progress of a typical reduction is shown in Fig. 1, in which, for purposes of clarity, the cobalt content of the solution is magnified ten times.

The operating procedure which has been found to be efficient in the pilot plant comprises the initial precipitation of fine particles of nickel with hydrogen, followed by particle growth in subsequent reductions from purified solutions in a semi-continuous process.

The purified solution is charged to an autoclave, the temperature is raised to approximately 350° F., and hydrogen is introduced at an overpressure of about 400 lb./in.<sup>2</sup>, the total pressure being about 500 lb./in.<sup>2</sup>. With proper agitation, the soluble nickel is completely reduced to metal particles about 1  $\mu$  in dia. in 15–20 min. (see Figs. 2 and 3). In the pilot plant, operation is stopped when the nickel content of the solution is reduced to about 1 g./l., the cobalt remaining at about 0.7 g./l. At this time the agitators are stopped, the nickel is allowed to settle for 3–5 min., and the supernatant solution discharged to a blow-off tank at atmospheric pressure.

A second charge of purified solution is introduced into the

autoclave, the agitators started, the temperature raised, and hydrogen introduced as before. The nickel particles present in the solution provide a large surface area upon which the new nickel is precipitated. The particles are again allowed to settle and the supernatant liquid is discharged. The nickel reduced in this step is deposited on the finely divided nickel particles from the previous step.

This reduction procedure is repeated until the nickel powder particles in the autoclave have grown to about 50  $\mu$  in dia. and nickel metal is present in an amount of 500–800 g./l. The autoclave is then discharged with the agitators in operation, the nickel and depleted solution both going to a blow-off tank, where they are separated by gravity. This series of operations is carried out repeatedly, a charge of nickel being removed at suitable intervals.

As the reduction operation continues, a small amount of nickel tends to adhere to the walls and agitators, owing, possibly, to its tendency to plate on other metal surfaces.

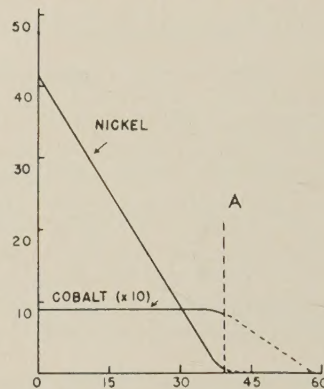
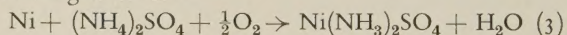


FIG. 1.—Nickel Precipitation by Hydrogen.

A = cut-off point for Ni precipitation.

When the amount of adhering nickel is sufficient to justify its removal, the charge is removed from the autoclave, the hydrogen purged, and a solution of ammonium sulphate introduced; the agitators are then started again and the vessel pressurized with compressed air. In a 6-hr. leaching period, the nickel is dissolved from the vessel walls according to the following reaction:



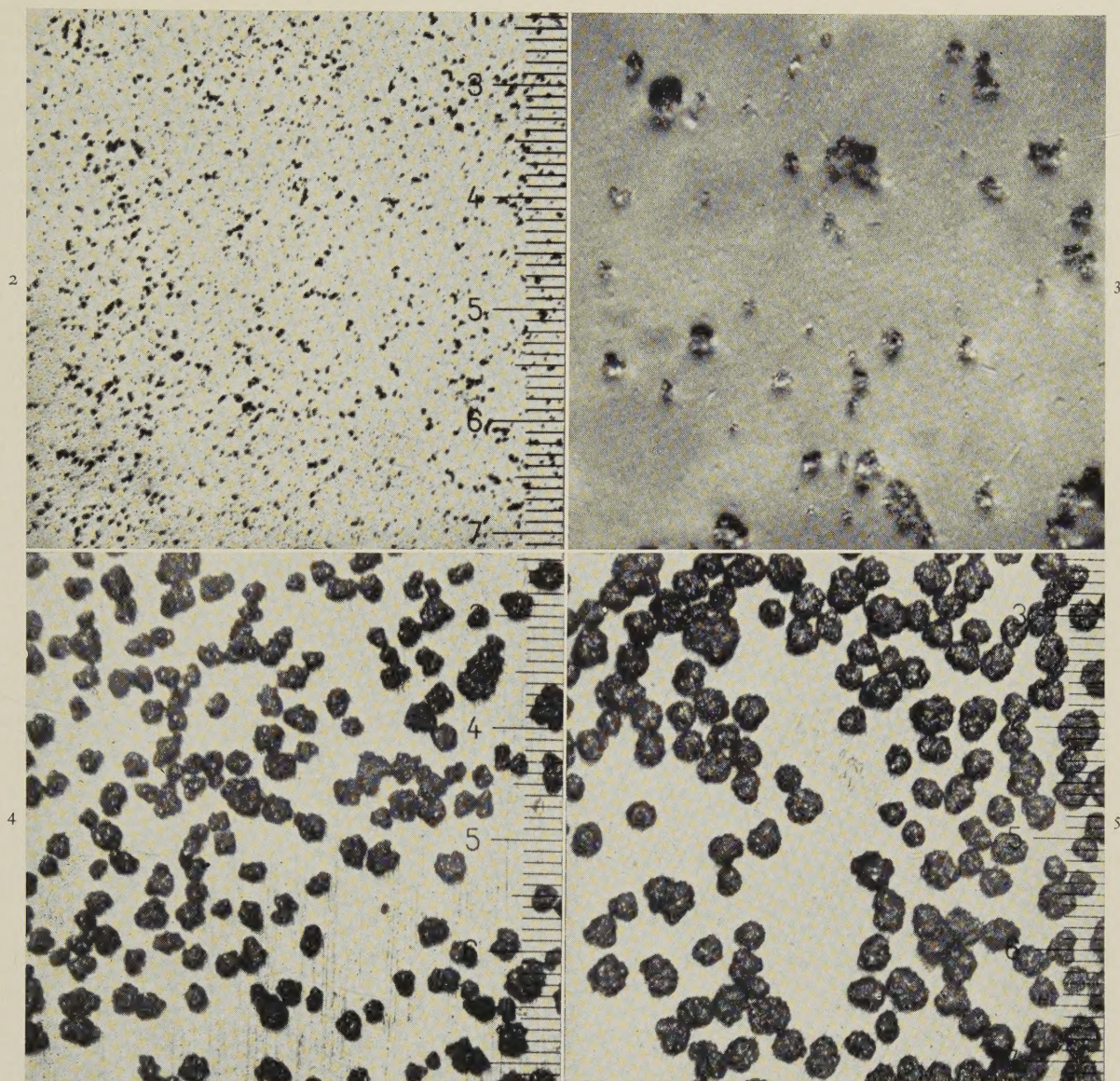
thus reproducing the nickel ammine, which can be reduced with hydrogen to produce finely divided nickel particles, and the sequence of operations is then repeated.

The nickel powder recovered from the blow-off tank is washed with water to remove ammonium salts, after which it is steam-dried and packed in containers ready for use.

The supernatant solution containing about 1 g./l. of nickel and 0.7 g./l. of cobalt is treated with H<sub>2</sub>S to precipitate the two metals as mixed nickel-cobalt sulphide, leaving a pure ammonium sulphate solution containing about 400 g./l. ammonium sulphate, which is then evaporated to produce a fertilizer-grade product. The nickel-cobalt sulphide is re-leached, the nickel and cobalt separated, and the cobalt finally recovered in an ammine solution, which, like the nickel, is treated with hydrogen at elevated temperature and pressure to produce pure cobalt powder.

### Properties of the Powder

Figs. 2–5 show clearly the character of the nickel powder produced at different periods in the cycle. The uniform particle size of the final product (Fig. 5) is of particular interest.



FIGS. 2-5.—Nickel Powder Produced by Hydrogen Reduction of Ammine Solution.

FIG. 2.—Initial Stage.  $\times 110$ .FIG. 4.—Twelfth Stage.  $\times 110$ .FIG. 3.—Initial Stage.  $\times 860$ .FIG. 5.—Twenty-Seventh Stage.  $\times 110$ .

Fig. 3 taken at  $\times 860$  (compare with Fig. 2 taken at  $\times 110$ ) shows the spheroidal shape of the fine nickel particles produced in the initial stage.

A typical sample of the nickel powder produced in the pilot plant had the following percentage analysis:

Ni + Co	Co	Fe	S	Cu	Pb	C
99.97	0.10	0.01	0.012	0.002	<0.002	nil

Other metals were not detected (<0.001%).

Nickel powder with low-cobalt content is produced by re-leaching the regular powder product with ammonium sulphate and compressed air to produce a low-cobalt solution from which the nickel is reprecipitated with hydrogen. The low-cobalt powder analyses (per cent):

Ni + Co	Co	Fe	S	Cu	Pb	C
99.98	0.01	0.002	0.007	<0.002	<0.002	nil

Other metals were not detected (<0.001%).

The apparent density, particle size, and analysis of a number of representative samples from different stages of a complete cycle are given in the following table:

Stage Number	Apparent Density, g./c.c.	Average Diameter, $\mu$	Ni + Co Content, (by diff.), %	Co Content, %
1	1.012	—	—	—
2	1.808	—	99.81	0.08
6	3.008	31.7	99.91	0.09
12	3.862	35.6	99.96	0.11
20	4.120	49.9	99.98	0.10
27	3.830	51.0	99.97	0.09

If small amounts of impurities are not detrimental, it is possible, by controlling the conditions of reduction and removing the powder at a suitable stage, to produce a final

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product having a particle diameter of 1-150  $\mu$  and larger, and an apparent density from 0.25 to about 4.0 g./c.c., as desired. For special experimental use, a considerable quantity of powder has been produced to meet strict chemical specifications with iron content less than 0.05, sulphur content less than 0.01, and which consists entirely of particles between 3 and 10  $\mu$ . Techniques are available for producing in quantity nickel powders that will meet a wide variety of specifications.

The 50- $\mu$  powder produced in the pilot plant has been tested in electric furnaces producing stainless steel, and has been found to give very satisfactory results with no dusting or other mechanical loss of nickel. The powder melts readily in arc- and induction-furnaces provided that a small amount of solid or molten metal is present to initiate the melting action. Admixture of different-sized powders in suitable proportions permits the production of compacts.

### Potential Uses

The availability of high-purity nickel in powder form suggests many possible new uses. It can be used for porous sintered compacts such as battery plates; it dissolves readily in molten cast iron and can be added as a fine stream; it dissolves very rapidly in acid, and thus can provide a useful starting point for nickel salt production. It is a convenient material for research workers who wish to prepare nickel alloys. It can be rolled to produce pure nickel sheets with normal physical properties.

To summarize, it may be said that high-purity nickel can be produced by the combined leaching and hydrogen-precipitation process; the operations are relatively simple, straightforward, and easy to control; the nickel-reduction procedure is rapid; the reductant is comparatively inexpensive; labour requirement is low; and pure nickel powders can be produced to meet a wide variety of particle-size specifications.

### Acknowledgements

Grateful acknowledgement is made to the many people who, by their efforts, have made the new process possible, and to Sherritt Gordon Mines Ltd. and the Chemical Construction Corporation for their generosity in granting permission to publish the data given in this paper. The author is indebted to Mrs. W. M. Armstrong for preparing the photomicrographs.

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**IMPERIAL CHEMICAL INDUSTRIES, LTD.**, Metals Division, invites applications from Metallurgists, Chemists, Physicists, and Engineers (mechanical and electrical) to fill vacancies as Technical Officers and Assistant Technical Officers in its Research and other Departments in Birmingham and elsewhere. Metallurgists and Chemists are required principally for industrial or fundamental research work associated with THE DEVELOPMENT OF THE NEW METAL TITANIUM. Physicists and Engineers are required for development work in other fields. Candidates for the Technical Officer grade must possess either a good honours degree or the equivalent professional qualification: those for the Assistant Technical Officer grade must possess a pass degree, Higher National Certificate, or the equivalent. For the Research posts, previous experience would be advantageous, though not essential. Suitably qualified men serving in H.M. Forces will be considered provided they will be available for employment during 1954. Commencing salaries will depend on age, qualifications, and experience, and will progress by annual increments to £1200 per annum for Technical Officers and £850 per annum for Assistant

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**METALLURGIST** required for research and development work on the production of magnesium alloy castings. Applicants should possess Hons. degree in metallurgy or A.I.M. Initiative, ability to work with minimum supervision, and experience in castings technology essential. Address applications in writing, stating age, qualifications, and experience, to Personnel Manager, Magnesium Elektron, Ltd., Clifton Junction, near Manchester.

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**RESEARCH LABORATORIES** of The General Electric Co., Ltd., North Wembley, Middx., have the following vacancies for graduates: Metallurgists, Physicists, and Physical Chemists for research and development work in the Metallurgy Group in the following fields: (1) Powder Metallurgy. (2) Brazing. (3) Controlled atmospheres. (4) General investigations (non-ferrous metals). Candidates should be not older than 30 years of age, and if under 26 must have completed National Service. Apply in writing to Staff Manager, (Ref. RLO/313), giving full particulars of age, qualifications, and experience.

**SENIOR METALLURGIST** required, preferably between 30 and 35 years of age, with at least Second Class Honours Degree or equivalent. This is a responsible and progressive position with an appropriate salary. Apply, Personnel Manager, McKechnie Brothers, Ltd., Rotton Park Street, Birmingham 16.

**TECHNICAL ASSISTANT** required for Manager of London Factory engaged on manufacture of sintered hard metal. Applicants should have had practical experience in the powdered metals industry, coupled with engineering qualifications, initiative, and ability to handle administrative matters. Salary dependent on qualifications and experience. Write giving full details and salary required to Box No. 364, The Institute of Metals, 4 Grosvenor Gardens, London, S.W.1.

**THE MORGAN CRUCIBLE CO., LTD.**, will be making a Technical Sales appointment. Applications are invited from men 28-35 with a metallurgical and foundry background, experience of sales office administration, and the ability to collaborate with technical staff. Specialist training will be given in crucible, furnace, and refractory fields. The position is permanent, pensionable, and carries good prospects. Write giving details of age, experience, and qualifications to the Staff Manager, Battersea Church Road, London, S.W.11.

### UNIVERSITY OF BIRMINGHAM

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Applications should reach the Registrar, The University, Edgbaston, Birmingham 15, as soon as possible.

Further particulars may be obtained from the undersigned.

The University, Birmingham 3.

C. G. BURTON,  
Secretary.